

AD-A158 346

THE EFFECTS OF MIL-R-81294 PAINT STRIPPER ON AS/3501-6
GRAPHITE/EPOXY COM. (U) NAVAL AIR DEVELOPMENT CENTER
HARMINSTER PA AIRCRAFT AND CREW S. S J SPADAFORA

1/1

UNCLASSIFIED

22 MAY 84 NADC-85003-60

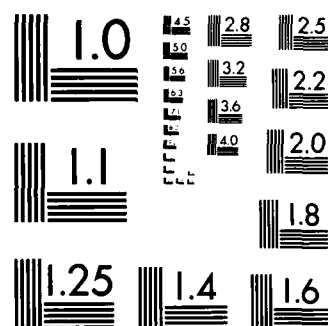
F/G 11/4

NL

END

FILED

ETC



MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS 1963 A

AD-A158 346

2

REPORT NO. NADC-85003-60



THE EFFECTS OF MIL-R-81294 PAINT STRIPPER ON AS/3501-6 GRAPHITE/EPOXY COMPOSITE SYSTEMS

Stephen J. Spadafora
Aircraft and Crew Systems Technology Directorate
NAVAL AIR DEVELOPMENT CENTER
Warminster, Pennsylvania 18974

22 MAY 1984

FINAL REPORT
AIRTASK NO. AF61-542-000
WORK UNIT ZM540

Approved for Public Release; Distribution Unlimited

DTIC FILE COPY

Prepared For
NAVAL AIR SYSTEMS COMMAND
Department of the Navy
Washington, DC 20361

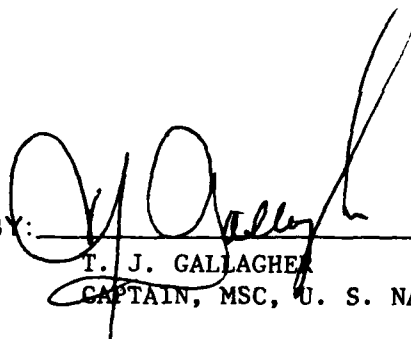
85 8 23 00 4

NOTICES

REPORT NUMBERING SYSTEM – The numbering of technical project reports issued by the Naval Air Development Center is arranged for specific identification purposes. Each number consists of the Center acronym, the calendar year in which the number was assigned, the sequence number of the report within the specific calendar year, and the official 2-digit correspondence code of the Command Office or the Functional Directorate responsible for the report. For example: Report No. NADC-78015-20 indicates the fifteenth Center report for the year 1978, and prepared by the Systems Directorate. The numerical codes are as follows:

CODE	OFFICE OR DIRECTORATE
00	Commander, Naval Air Development Center
01	Technical Director, Naval Air Development Center
02	Comptroller
10	Directorate Command Projects
20	Systems Directorate
30	Sensors & Avionics Technology Directorate
40	Communication & Navigation Technology Directorate
50	Software Computer Directorate
60	Aircraft & Crew Systems Technology Directorate
70	Planning Assessment Resources
80	Engineering Support Group

PRODUCT ENDORSEMENT – The discussion or instructions concerning commercial products herein do not constitute an endorsement by the Government nor do they convey or imply the license or right to use such products.

APPROVED BY: 
T. J. GALLAGHER
CAPTAIN, MSC, U. S. NAVY

DATE: 17 January 1985

✓

AH

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION			1b. RESTRICTIVE MARKINGS		
2a. SECURITY CLASSIFICATION AUTHORITY			3. DISTRIBUTION/AVAILABILITY OF REPORT		
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE			Approved for Public Release; Distribution Unlimited.		
4. PERFORMING ORGANIZATION REPORT NUMBER(S) NADC-85003-60			5. MONITORING ORGANIZATION REPORT NUMBER(S)		
6a. NAME OF PERFORMING ORGANIZATION Naval Air Development Center		6b. OFFICE SYMBOL (If applicable)		7a. NAME OF MONITORING ORGANIZATION	
6c. ADDRESS (City, State, and ZIP Code) Aircraft and Crew Systems Technology Directorate Warminster, PA 18974			7b. ADDRESS (City, State, and ZIP Code)		
8a. NAME OF FUNDING/SPONSORING ORGANIZATION Naval Air Systems Command		8b. OFFICE SYMBOL (If applicable)		9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER	
8c. ADDRESS (City, State, and ZIP Code) Washington, DC 20361			10. SOURCE OF FUNDING NUMBERS		
			PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO. See Reverse Side
			WORK UNIT ACCESSION NO. ZM540		
11. TITLE (Include Security Classification) The Effects of MIL-R-81294 Paint Stripper on AS/3501-6 Graphite/Epoxy Composite Systems					
12. PERSONAL AUTHOR(S) Stephen J. Spadafora					
13a. TYPE OF REPORT Final		13b. TIME COVERED FROM TO		14. DATE OF REPORT (Year, Month, Day) 22 May 1984	
15. PAGE COUNT					
16. SUPPLEMENTARY NOTATION					
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD	GROUP	SUB-GROUP			
			Graphite/Epoxy Composites		
			Phenolic MIL-R-81294 Paint Stripper		
			Nonphenolic MIL-R-81294 Paint Stripper		
19. ABSTRACT (Continue on reverse if necessary and identify by block number)					
<p>The use of resin matrix composites for structural applications on naval aircraft has dramatically increased over the past decade. Nine percent of the F-18 Hornet is AS/3501-6 graphite/epoxy composite material. This composite material is predominantly surface structure and is coated with the standard paint scheme (MIL-P-23777 epoxy primer and MIL-C-83286 polyurethane topcoat). During general aircraft rework operations, the coating system of an aircraft is chemically stripped, which would result in the case of the F-18 to exposure of composite material to paint stripper. Previous studies have shown that the standard paint stripper MIL-R-81294 has an adverse effect on the epoxy matrix of these composite systems under total immersion exposure conditions. NAVAIRDEVGEN was tasked to evaluate the effects of MIL-R-81294 paint stripper on AS/3501-6 graphite/epoxy composites under simulated rework conditions.</p> <p>Results of this study show that MIL-R-81294 causes a statistically significant decrease in the physical properties of AS/3501-6 composite materials under simulated rework conditions. This deleterious effect was concentrated at the composite surface directly exposed to the paint stripper. (continued)</p>					
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT <input type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT <input type="checkbox"/> DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION		
22a. NAME OF RESPONSIBLE INDIVIDUAL			22b. TELEPHONE (include Area Code)		22c. OFFICE SYMBOL

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE

Block No. 10:

AIRTASK NO. 310-310A/001B/4F61-542-000

Block No. 19:

AS/3501-6 graphite/epoxy composite structures should not be exposed to MIL-R-81294 paint stripper.

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE

TABLE OF CONTENTS

	<u>Page</u>
LIST OF TABLES.....	ii
LIST OF FIGURES.....	ii
INTRODUCTION.....	1
MATERIALS	1
PROCEDURES.....	2
Exposure.....	2
Nondestructive Testing (NDT)	2
Tensile Test.....	2
Four Point Flexure Test	2
Dynamic Mechanical Analysis	2
Wedge Crack Extension Test.....	3
Creep Test.....	3
Fatigue Test	3
RESULTS AND DISCUSSION	3
Nondestructive Inspection.....	3
Tensile Test.....	4
Four Point Flexure Test	4
Dynamic Mechanical Analysis (DMA)	4
Wedge Crack Extension Test	4
Creep Test.....	5
Fatigue Test	5
General Discussion	5
CONCLUSIONS	6
RECOMMENDATIONS	6
REFERENCES.....	6
ACKNOWLEDGEMENT.....	6
APPENDIX A	
Graphite/Epoxy AS/3501-6 Prepreg Properties.....	A-1
Cure Cycle for AS/3501-6 Graphite/Epoxy Composite System.....	A-1

LIST OF TABLES

	<u>Page</u>
I. Exposure Conditions	7
II. (a) Room Temperature Tensile Test Results	7
(b) 180°F Tensile Test Results	8
III. (a) Room Temperature Four Point Flexure Test Results	9
(b) 180°F Four Point Flexure Tests Results	10
IV. Glass Transition Temperatures From DMA	11
V. Wedge Crack Growth With Subsequent Strain Energy Release Rate Changes (ΔG_I)	12
VI. Creep Test Residual Tensile Strength Results	13
VII. Fatigue Test Results	13
VIII. Statistical Significance of Test Results	14

LIST OF FIGURES

	<u>Page</u>
1) Wedge Crack Test Specimen	15
2) Wedge Crack Growth Rate	16
3) Creep Test Results (50% Load)	17
4) Creep Test Results (40% Load)	18

INTRODUCTION

Over the past decade, naval aircraft have been constructed with more and more resin matrix composites. The F-4 Phantom II contains essentially no composite material while the F-18 Hornet and AV-8B Harrier contain 9% and 26% respectively. The graphite/epoxy composite material on the F-18 is predominately surface structure and is coated with the standard paint scheme. Periodic reworking was not originally scheduled for the F-18 according to NARF personnel (reference (A)), although several have already been brought in for repairs at NARF North Island.

During current rework operations, the coating system of the aircraft is chemically stripped, resulting in exposure of the composite material to paint stripper in the case of F-18. Previous studies (references (B) and (C)) have shown that the standard epoxy/polyurethane paint stripper MIL-R-81294 has an adverse effect on the epoxy matrix of these composite systems. One study (reference (B)) evaluated the quantitative deleterious effects of the MIL-R-81294 paint stripper on AS/3501-6 graphite/epoxy composites under total immersion conditions. The qualitative effects of MIL-R-81294 on a graphite/epoxy S-3A lower composite spoiler under controlled laboratory exposure conditions were reported in reference (C). In this study sections of an S-3A graphite/epoxy composite spoiler were exposed to MIL-R-81294 paint stripper and then tested using Dynamic Mechanical Analysis (DMA). The results of these tests showed a qualitative degradation of the matrix, but the specific amount of degradation could not be determined with this method. Until now there has not been a study of this phenomenon under simulated rework exposure conditions.

In this investigation (performed under airtask #AIR-310-310A/001B/4F61-542-000 work unit #ZM540), graphite/epoxy composite panels were exposed to chemical paint strippers under various time and temperature conditions. The panels were then rinsed with water, dried and nondestructively evaluated to determine any anomalies in the material due to exposure like dissolved matrix or resin/fiber debonds. Subsequently, specimens were cut from the exposed material and the following series of mechanical tests were performed.

- 1) Tensile
- 2) Fatigue
- 3) Creep
- 4) Four Point Flexure
- 5) Dynamic Mechanical Analysis
- 6) Wedge Crack Extension

MATERIALS

The composite material used in this investigation was the Hercules AS4/3501-6 graphite fiber/epoxy matrix system. Panels of this material were fabricated using three laminate layups: 8 ply symmetric ($\pm 45^\circ$)_S, 13 ply ($O_2, \pm, O_2, 90, O_2, \pm, O_2$) and 50 ply symmetric ($O, \pm, O_2, \pm, O_2, \pm, O, 90, O, \pm, O_2, \pm, O_2, \pm, O$)_S. The prepreg properties of this material and the cure schedule for these panels are listed in Appendix A. Following the cure of these panels, they were nondestructively examined to determine any anomalies due to fabrication such as voids or fiber discontinuities.

The chemical paint strippers used in this investigation were type I phenolic and type II non-phenolic versions of MIL-R-81294 epoxy-polyurethane paint stripper.

PROCEDURES

EXPOSURE

Graphite/epoxy test panels were dried in an oven in order to eliminate any moisture absorbed following post-cure. This was accomplished by periodically weighing the panels until a constant weight was obtained. After drying, the peripheries of the panels were masked in order to eliminate the possibility of any edge effects. Exposure consisted of placing panels horizontally and pouring the paint stripper onto the surface of the panels to form a layer approximately one eighth inch (0.32 cm) thick. The temperature and time conditions of exposure are listed in Table I. Following the exposure period, paint stripper was removed by rinsing with tap water and then drying the panels for 24 hours at room temperature.

NONDESTRUCTIVE TESTING (NDT)

Following paint remover exposure, the composite panels were nondestructively examined using an Impactoscope and A-Scan Ultrasonics. The Impactoscope is an automated taptester that applies a slight impact to the surface of the specimen and records the absorbed energy versus time response resulting from the impact. As abnormalities of the specimen are encountered, the response changes as indicated by shifts in the amplitude and horizontal position of the recorded wave form. In A-scan ultrasonics, the pulse-echo mode of an Ultrasonic Flaw Detector Set (AN/GSM-238) was used. With this method, an ultrasonic signal is applied to the surface of the panel and the return echo from the back of the panel is measured. As abnormalities in the panel are encountered, the return echo appears at a different location on the display screen of the test equipment.

TENSILE TEST

Tensile tests were performed as defined in ASTM #D 3039-76, Standard Test Method for Tensile Properties of Fiber-Resin Composites. One inch by eight inch (2.54 by 20.32 cm) tensile specimens were cut from eight ply panels. These specimens were not fitted with end tabs as recommended in the ASTM method, since a two inch (5.08 cm) grip area on each end was sufficient to prevent failure in the grips. An Instron Test Machine Model TT-D series #3298 was used for this test with a crosshead speed of 0.05 inches per minute (0.127 centimeters per minute). Five replicates from each exposure condition were tested at $72 \pm 5^\circ\text{F}$ ($22 \pm 3^\circ\text{C}$) and four or five replicates from each exposure condition were tested at $180 \pm 5^\circ\text{F}$ ($82 \pm 3^\circ\text{C}$).

FOUR POINT FLEXURE TEST

Four point flexure tests were performed in accordance with ASTM #D790-70, Standard Methods of Test For Flexural Properties of Plastics. One inch by three inch (2.54 by 7.62 cm) flexure specimens were cut from the thirteen ply panels. These specimens were approximately one sixteenth inch (0.16 cm) thick and had a two inch (5.08 cm) load span (length to depth ratio = 32:1). Testing was performed using an Instron Test machine and a crosshead speed of 0.1 inches per minute (0.254 cm per min). Four or five replicates were tested for each exposure condition at both $72 \pm 5^\circ\text{F}$ ($22 \pm 3^\circ\text{C}$) and $180 \pm 5^\circ\text{F}$ ($82 \pm 3^\circ\text{C}$).

DYNAMIC MECHANICAL ANALYSIS

Dynamic mechanical analysis was performed with a Dupont 981 Dynamic Mechanical Analyzer. One quarter inch by one inch (0.635 cm by 2.54 cm) specimens were cut from the thirteen ply test panels. Two replicates were tested from each exposure condition.

WEDGE CRACK EXTENSION TEST

The wedge crack test was developed for graphite/epoxy composite material by modifying ASTM #D3762-79, Standard Test Method For Adhesive-Bonded Surface Durability of Aluminum (Wedge Test). Using the properties of the aluminum specimen in the test method, a one inch by six inch (2.54 cm by 15.24 cm) graphite/epoxy wedge crack test specimen was designed as shown in figure 1. This test specimen was cut from the 50 ply panel each half of which was symmetric. A one-eighth inch (0.32 cm) thick stainless steel wedge was inserted between the 0° plies at the center of the specimen to induce the initial crack in the matrix. The test was conducted at 95°F (35°C) as in the ASTM method with the specimen totally immersed in the test fluid. Crack length measurements were made after 1, 4, 24, 48, 120 and 192 hours of exposure. Specimens were then rinsed with tap water and exposed to 140°F (60°C) and 100% relative humidity for one week. The final crack length measurements were made after this additional exposure period as is the practice with adhesively-bonded aluminum test specimens.

CREEP TEST

The creep test in this investigation was a tensile creep test based on ASTM #D2990-77, Standard Test Method for Tensile, Compressive and Flexural Creep and Creep-Rupture of Plastics. One inch by five inch (2.54 cm by 12.7 cm) creep specimens were cut from the eight ply panels, to which one inch (2.54 cm) square bare 7075 aluminum end tabs were bonded with an epoxy adhesive. Specimen gage length was three inches (7.62 cm). Strain gages were mounted on both the side exposed to paint remover and the unexposed side of the test specimen to monitor changes in strain rate. The tests were conducted at room temperature. Stresses used during this test were 25%, 33-1/3%, 40% and 50% of the ultimate tensile strength. The duration of each test was one week with two specimens tested at each stress level. After the creep tests were completed, the specimens from the 50% load condition were tested for residual tensile strength at room temperature using the procedure from the Tensile Test section.

FATIGUE TEST

The fatigue test for this investigation was a tension-tension fatigue test conducted in accordance with ASTM #D3479-76, Standard Test Method for Tension-Tension Fatigue of Oriented Fiber-Resin Matrix Composites. One inch by nine inch (2.54 cm by 22.86 cm) test specimens were cut from the eight ply panels and fitted with two inch (5.08 cm) long end tabs, leaving a five inch (12.7 cm) test gage length. The end tabs were eight ply glass/epoxy composite with a (0°, 90°) Symmetric layup, bonded to the specimens with an epoxy adhesive. Testing was performed with a MTS Material Test System 22 Kip load frame and a MTS model 810 control system. A stress ratio of ten to one was used, with the specimen being cyclically loaded between 6% and 60% of its estimated ultimate tensile strength. The fatigue test was performed at a frequency of 600 cycles per minute. Seven replicates per condition were tested at room temperature.

RESULTS AND DISCUSSION

NONDESTRUCTIVE INSPECTION

Impactoscope and A-scan ultrasonics readings varied over the surface of all test panels making it impossible to differentiate between the different test specimens. The variance in these readings was probably due to preexistent anomalies inherent in the nature of these materials. Nondestructive inspection methods used in this investigation were chosen because they represent typically available fleet inspection equipment.

TENSILE TEST

The tensile tests were designed to demonstrate effects on the entire matrix of the test specimen. Any significant chemical attack on the matrix would be seen as a change in the ultimate tensile strength of the specimen. The results of the tensile tests appear in Tables II(a) and II(b). Room temperature tests results fell within $\pm 2\%$ of the average value for the control specimen, showing no significant differences between exposed and unexposed specimens. Results for the 180°F (82°C) tests indicate a slight difference for some of the exposure conditions. These slight differences were statistically significant in the 95% confidence level, however, since they were in the range of 4% to 6-1/2%, they may not have any real engineering significance.

FOUR POINT FLEXURE TEST

The four point flexure test was designed to detect effects on the surface layers of the composite. The top surface of the test specimen is in compression while the bottom surface of the test specimen is in tension. Since graphite/epoxy composites are more sensitive to compressive failure than tensile failure, the exposed surface of the specimen was tested in the compression mode (top surface). Results of the four point flexure tests appear in Tables III(a) and III(b). Room temperature flexure tests showed that some of the test specimens had a statistically significant decrease in flexural strength at the 95% confidence level. These decreases, typically 4 to 6 percent, occurred in all of the exposure conditions at 110°F (43°C) except for one of the nonphenolic conditions which had a large standard deviation. Results for 180°F (82°C) flexure tests also showed statistically significant decreases for some of the specimens, including all of the phenolic exposures at 110°F (43°C). Flexural strength losses ranged from 3-1/2% to 10-1/2%. In all cases, the average flexural strength values for the exposure conditions fell below the average values of the control specimens.

DYNAMIC MECHANICAL ANALYSIS (DMA)

The dynamic mechanical analysis test measures the ability of a material to store and dissipate mechanical energy on deformation. DMA measured properties, known as storage and loss modulus, are temperature dependent. A material attains its maximum damping at its glass transition temperature, which is the temperature for the onset of segmental molecular motion. Gross chemical changes to the material should result in shifts of the glass transition temperature or the appearance of additional peaks in the moduli curves. The DMA testing resulted in almost identical curves for all specimens. There were no significant shifts or additional peaks for any exposure condition and the glass transition temperatures were relatively constant as shown in Table IV.

WEDGE CRACK EXTENSION TEST

The wedge crack test was the only test in this investigation in which the specimen was stressed during exposure. The crack growth results and subsequent changes in strain energy release rate are shown in Table V. These results show that the air and water controls had virtually no growth (less than 0.01 inch (0.0254 cm)) after eight days immersion and one additional week of exposure to 100% R.H. and 140°F (60°C). Paint stripper immersed specimens, however, had considerable growth due to exposure. After rinsing with water and further exposure to 100% R.H. and 140°F (60°C) for one week, the cracks continued to grow as if they were still immersed in paint stripper as shown in figure 2. Strain energy release rate for control specimens was nearly constant throughout the test, while strain energy release rates for type I and type II MIL-R-81294 exposed specimens were reduced by 56% and 45% respectively. Also, the test specimens immersed in the type I phenolic stripper exhibited blistering of the surface plies after 24 hours of immersion.

CREEP TEST

In the creep test, specimens were subjected to a constant tensile load to determine the exposure effects on the creep rate of the specimens. Only specimens exposed to phenolic remover at 110°F (43°C) for two, four-hour periods and control specimens were tested. The control and exposed specimens exhibited no significant differences under 25% and 33-1/3% load tests, but due to recorder malfunction these results could not be plotted directly. Creep test results for 40% and 50% of ultimate tensile strength load appear in figures 3 and 4. Residual tensile strength results for the 50% load specimens and the control specimens appear in Table VI. The graphs of strain versus time show virtually no difference for the 50% load and only a few percent difference for the 40% load. Residual tensile strength tests on creep specimens tested at 50% of U.T.S. indicated no difference between the controls and the exposed specimens.

FATIGUE TEST

In the tensile fatigue test, specimens were tested to determine the effect of exposure on the composite fatigue life. Results from the fatigue tests appear in Table VII. Specimens exposed to phenolic remover at 110°F (43°C) for two, four-hour periods and control specimens exhibited no significant difference in fatigue life. Since these two extreme conditions were not statistically different, no other conditions were tested.

GENERAL DISCUSSION

Tests results from the investigation indicate there is a statistically significant effect on the graphite/epoxy composite material due to the paint stripper exposure. A summary of the tests conducted and their relative statistical significance appears in Table VIII. The greatest effects from exposure were seen in the four point flexure tests and the wedge crack tests. In four point flexure, the test specimen has the maximum stress concentrated in the outer plies, where any chemical attack would most likely occur, while the wedge crack test emphasizes the effect of the paint stripper at a crack tip. Wedge crack tests also showed outer surface blistering due to exposure to phenolic paint stripper as well as continued crack growth even after the paint stripper was rinsed from the specimen.

Since any losses of mechanical properties were concentrated at surfaces in contact with paint stripper, the surface characteristics and refinishing properties of the composite material after exposure to paint stripper should be investigated. As an initial investigation into this phenomenon, AS/3501-6 graphite/epoxy panels were exposed to both types of MIL-R-81294 for two, four-hour exposures at 110°F (43°C) and then rinsed with tap water and dried for 24 hours at room temperature. The exposed panels were then coated with the standard naval aircraft paint system (MIL-P-23377 epoxy/polyamide primer and MIL-C-83286 polyurethane topcoat). After a one week air dry cure time, these test panels were sent out for one year of exposure to a carrier environment. The panels will be examined for blistering or removal of the coating system or other effects after carrier exposure.

This study did not consider several factors that may have altered or intensified the paint stripper effect on the graphite/epoxy composite material. These include such factors as: service environment conditions (such as moisture, UV radiation, SO₂ from exhaust gas, etc.), material quality (service damage, incomplete cure or preexistent flaws) and the effects of multiple cycles of reworking and in-service operations.

CONCLUSIONS

1. MIL-R-81294 paint stripper causes a statistically significant deleterious change in the physical properties, such as the loss in flexural strength, of AS/3501-6 graphite/epoxy composites under the exposure conditions in the investigation. This effect is concentrated at the surface that is in direct contact with the paint stripper.
2. AS/3501-6 graphite/epoxy composite structures should not be chemically paint stripped with MIL-R-81294 epoxy/polyurethane paint stripper.

RECOMMENDATIONS

1. Other methods of paint removal from graphite/epoxy should be investigated, such as plastic pellet paint stripping.

REFERENCES

- (A) Phoncon NAVAIREWORKFAC, NAS, North Island (Code 340), A. Bleich/NAVAIRDEVCCN (Code 6062) S. Spadafora of 4 May 1984.
- (B) Clark, K. G., "Compatibility of Aircraft Operational Fluids with a Graphite/Epoxy Composite — Development of an Exterior Coating System and Remover," NAVAIRDEVCCN Report No. NADC-80046-60, 26 June 1980.
- (C) Spadafora, S. J., "The Effects of MIL-R-81294 Paint Stripper on S-3A Lower Composite Spoilers," NAVAIRDEVCCN Technical Memorandum No. ACSTD-TM-2158, 22 June 1982.
- (D) Brown, S. R. and Pilla, G. J., "Titanium Surface Treatments for Adhesive Bonding," NAVAIRDEVCCN Report No. NADC-82032-60, 31 March 1982.

ACKNOWLEDGEMENT

The technical assistance of Mr. Kenneth Clark of the Materials Protection Branch throughout this investigation is greatly appreciated.

GRAPHITE/EPOXY AS/3501-6 PREPREG PROPERTIES

Resin Content	43%
Resin Flow	20%
Fiber Density	0.0655 Lbs/in ³
Fiber Areal Weight	2.2 x 10 ⁻⁴ Lbs/in ²

CURE CYCLE FOR AS/3501-6 GRAPHITE/EPOXY COMPOSITE SYSTEM

- 1) Pull vacuum on prepreg panel to greater than 25 inches (635 mm) Hg (mercury).
- 2) Raise temperature to 240°F (116°C) with vacuum at a rate of 3-5°F (1.7-3°C) per minute.
- 3) Hold at 240°F (116°C) and greater than 25 inches (635 mm) Hg for 1 hour.
- 4) Pressurize to 85 PSI (0.586 MPa) and raise temperature to 350°F (177°C) at 3-5°F (1.7-3°C) per minute.
- 5) Hold at 350°F (177°C), 85 PSI (0.586 MPa) and greater than 25 inches (635 mm) Hg for 2 hours.
- 6) Cool to 200°F (93°C) at 5°F (3°C) per minute.
- 7) Release vacuum and pressure, and cool to room temperature.
- 8) Post-cure panel at 350°F (177°C) and atmospheric pressure for 8-1/2 hours.

NADC-85003-60

APPENDIX

A

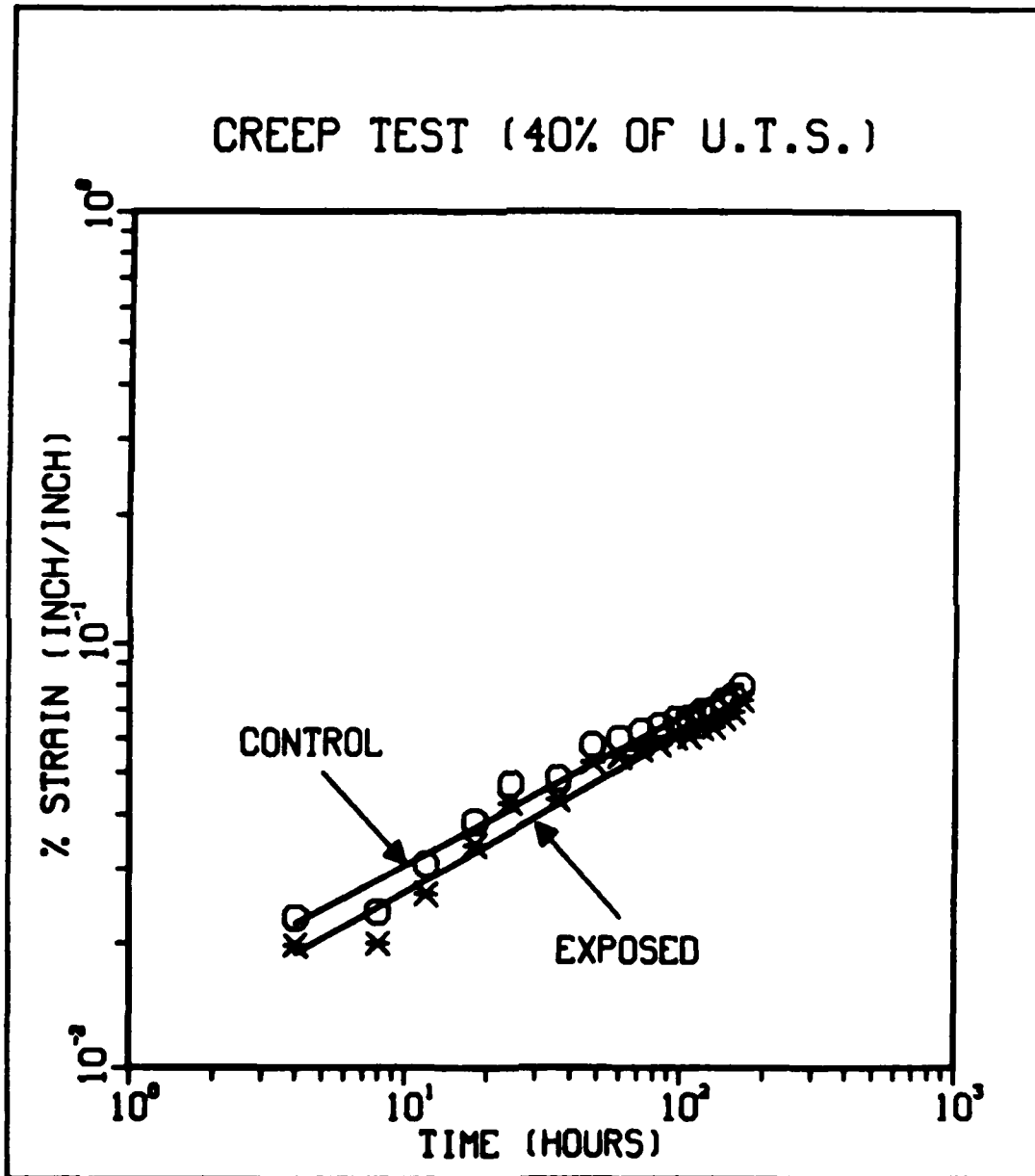


Figure 4. Creep Test Results (40% Load)

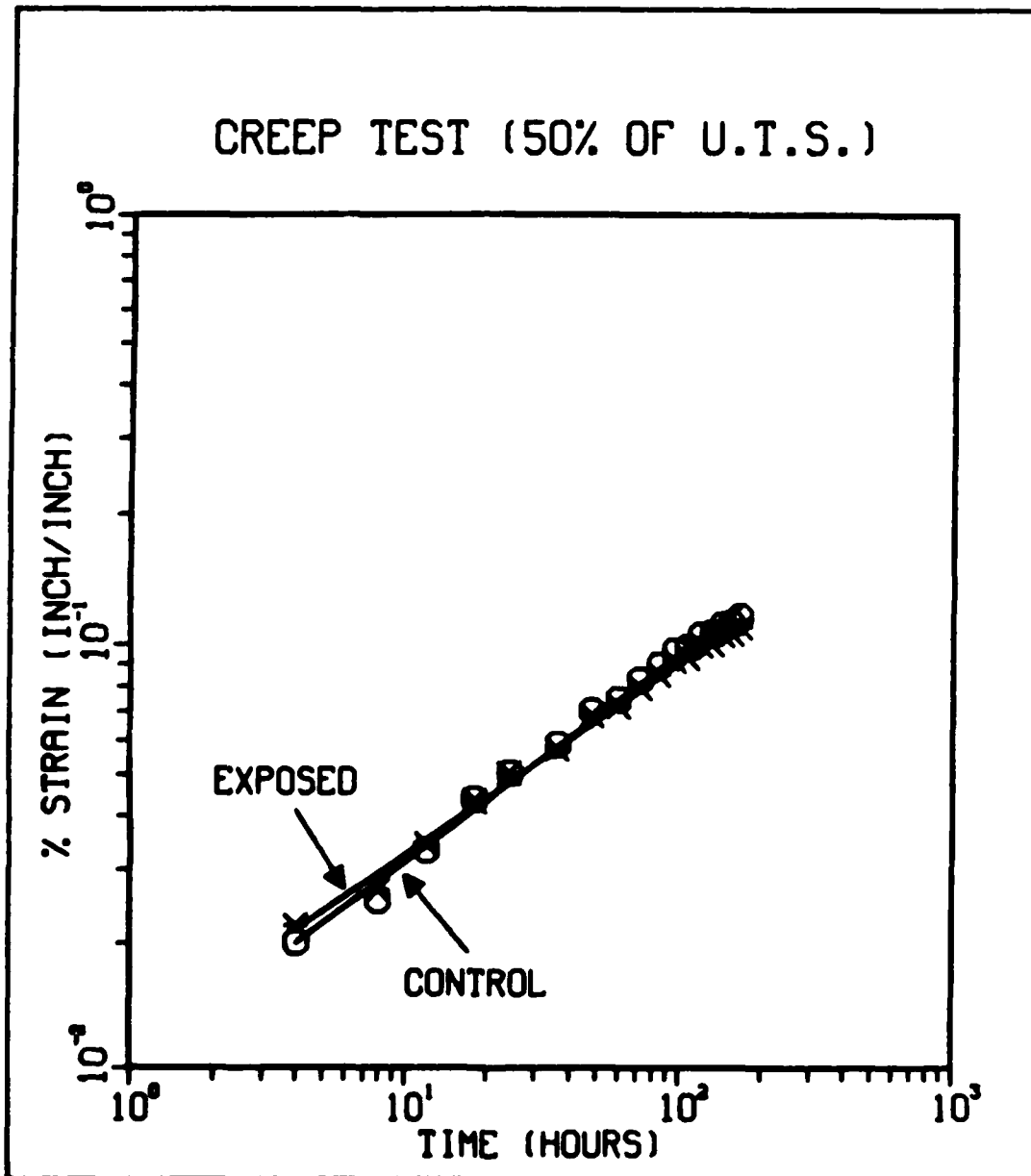


Figure 3. Creep Test Results (50% Load)

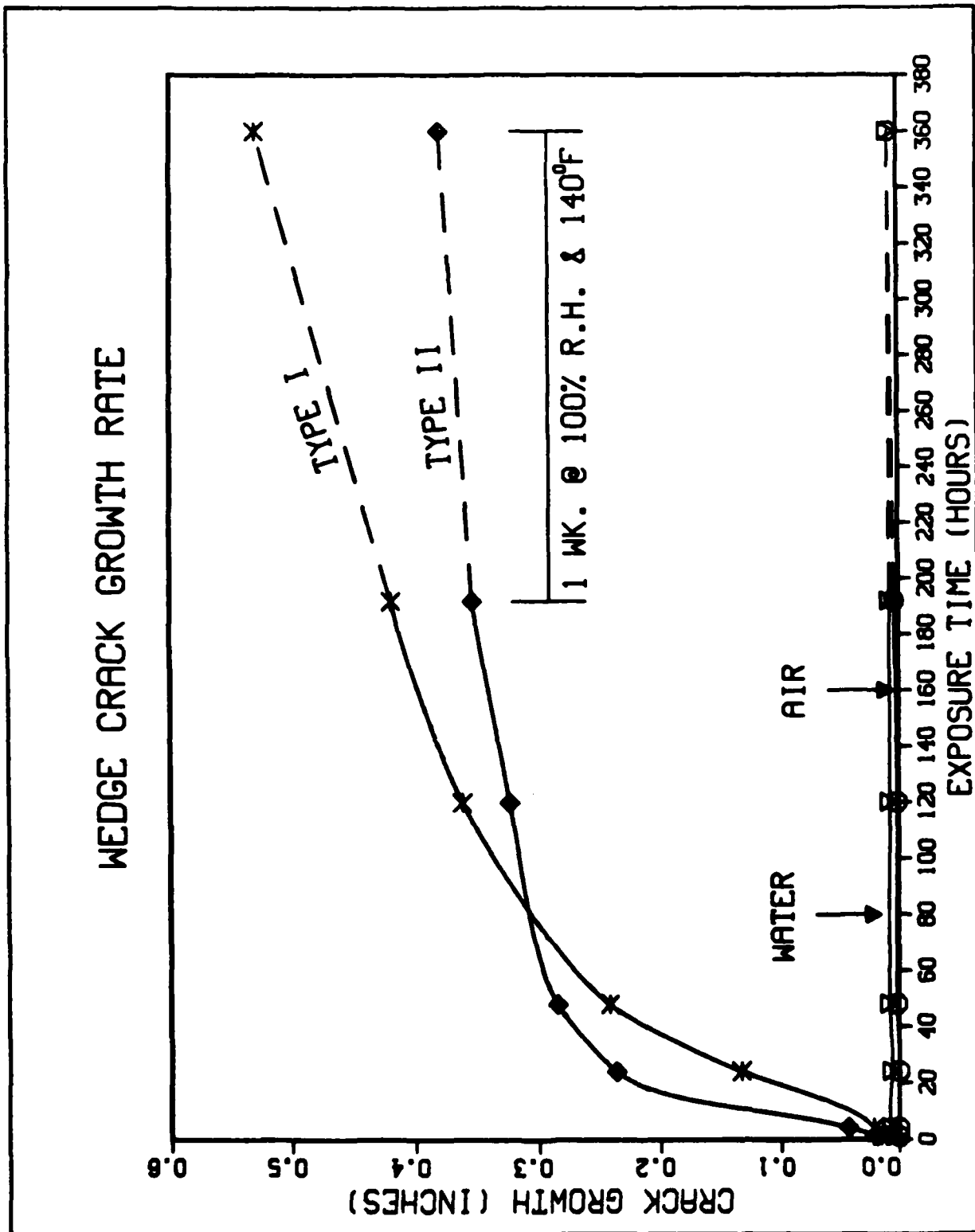
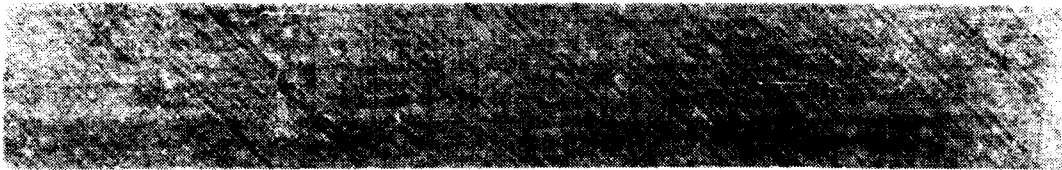
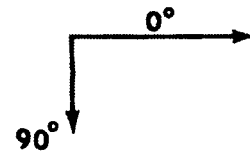


Figure 2. Wedge Crack Growth Rate

TOP VIEW



LAMINATE REFERENCE DIRECTION



SIDE VIEW



50 PLY AS/3501-6 GRAPHITE/EPOXY COMPOSITE MATERIAL

Figure 1. Wedge Crack Growth Specimen

TABLE VIII. STATISTICAL SIGNIFICANCE OF TEST RESULTS

Exposure Condition		Room Temp Tensile Test	180°F Tensile Test	Room Temp Four Point Flexure Test	180°F Four Point Flexure Test	Creep Test	Fatigue Test	Wedge Crack Test	DMA Test
<u>Phenolic</u>								Yes	
<u>Time</u>	<u>Temp</u>								
4	RT	Yes	No	No	No	—	—		No
4	110°F	Yes	Yes	Yes	Yes	—	—		No
8	RT	No	Yes	No*	No	—	—		No
8	110°F	Yes	No	Yes	Yes	—	—		No
4,4	RT	No	No	Yes	Yes	—	—		No
4,4	110°F	Yes	Yes	Yes	Yes	No	No*		No
<u>Nonphenolic</u>								Yes	
4	RT	No	No	No	No	—	—		No
4	110°F	No	Yes	No*	No	—	—		No
8	RT	No	No	Yes	Yes	—	—		No
8	110°F	Yes	Yes	Yes	No*	—	—		No
4,4	RT	No	No	No	Yes	—	—		No
4,4	110°	Yes	Yes	Yes	No*	—	—		No

* Large Standard Deviation

TABLE VI. CREEP TEST RESIDUAL TENSILE STRENGTH RESULTS

Exposure Condition			Residual Tensile Strength (PSI)	Standard Deviation (PSI)	Coefficient of Variation	% Loss	Statistical Significance
<u>Time</u>	<u>Type</u>	<u>Temp</u>					
	CONTROL		13800	71	0.51%	—	—
4,4	P	110°F	13800	106	0.77%	0.0%	No

TABLE VII. FATIGUE TEST RESULTS

Exposure Condition			Life Cycles To Failure	Standard Deviation	Coefficient of Variation	% Change	Statistical Significance
<u>Time (hrs)</u>	<u>Type</u>	<u>Temp</u>					
	CONTROL		560076	141345	25.2%	—	—
4,4	P	110°F	715121	175625	24.6%	+27.7%	No

TABLE V. WEDGE CRACK GROWTH WITH SUBSEQUENT STRAIN ENERGY RELEASE RATE CHANGES (ΔG_I)

Hours of Exposure	AIR		WATER (DISTILLED)		MIL-R-81294 TYPE I		MIL-R-81294 TYPE II	
	Growth	Total Length (inches)	Growth	Total Length (inches)	Growth	Total Length (inches)	Growth	Total Length (inches)
Initial	—	2.1933	—	2.3013	—	2.2425	—	2.2850
1	NG	2.1933	0.0038	2.3051	0.0150	2.2575	0.0188	2.3038
4	NG	2.1933	0.0025	2.3076	0.0063	2.2638	0.0238	2.3276
24	NG	2.1933	NG	2.3076	0.1125	2.3763	0.1938	2.5214
48	0.0013	2.1946	0.0013	2.3089	0.1075	2.4838	0.0495	2.5689
120	NG	2.1946	NG	2.3089	0.1200	2.6038	0.0388	2.6077
192	0.0025	2.1971	NG	2.3089	0.0575	2.6613	0.0300	2.6377
1 Week 100% RH 140°F	0.0038	2.2009	NG	2.3089	0.1100	2.7713	0.0263	2.6640
TOTAL	0.0076	2.2009	0.0076	2.3089	0.5288	2.7713	0.3790	2.6640
ΔG_I	0.0277	—	0.0220	—	1.0746	—	0.8001	—
$\Delta G_I\%$	-1.326%	—	-1.268%	—	-56.04%	—	-44.87%	—

NG = No Growth

 G_I = Strain Energy Release Rate Equation (Adapted from Ref. (D)). $G_I = 25.08(3(a + 0.0744)^2 + 0.0154)/((a + 0.0744)^3 + 0.0154a)^2$ Lbs/in²

a = distance from Load Point to Crack Tip (inches)

TABLE IV. GLASS TRANSITION TEMPERATURE FROM DMA

<u>Exposure Condition</u>			<u>T_g</u>
<u>Time</u>	<u>Type</u>	<u>Temp</u>	
	CONTROL		214°C
4	NP	RT	211°C
8	NP	RT	210°C
4,4	NP	RT	213°C
4	NP	110°F	216°C
8	NP	110°F	216°C
4,4	NP	110°F	215°C
4	P	RT	213°C
8	P	RT	214°C
4,4	P	RT	215°C
4	P	110°F	208°C
8	P	110°F	213°C
4,4	P	110°F	209°C

TABLE III(b). 180°F FOUR POINT FLEXURE TEST RESULTS

Exposure Condition			Flexural Strength (PSI)	σ_{N-1} (PSI)	Conv %	% Loss	95% Confidence Level Student T-Test Statistically Significant
<u>Time</u>	<u>Type</u>	<u>Temp</u>					
	CONTROL		210004	2087	0.99%	—	—
8	NP	110°F	206097	8548	4.15%	1.86%	No
4,4	NP	110°F	205717	12419	6.04%	2.04%	No
4	NP	RT	205522	5971	2.91%	2.13%	No
4	NP	110°F	204406	4770	2.33%	2.67%	No
8	Ph	110°F	202543	3896	1.92%	3.55%	Yes
4,4	NP	RT	202007	4378	2.17%	3.81%	Yes
8	Ph	RT	201109	6468	3.22%	4.24%	No
4,4	Ph	RT	200597	3530	1.76%	4.48%	Yes
4,4	Ph	110°F	200551	1111	0.55%	4.50%	Yes
4	Ph	RT	200376	10017	5.00%	4.58%	No
8	NP	RT	198914	6632	3.33%	5.28%	Yes
4	Ph	110°F	188226	10578	5.62%	10.37%	Yes

TABLE III(a). ROOM TEMPERATURE FOUR POINT FLEXURE TEST RESULTS

Exposure Condition			Flexural Strength (PSI)	σ_{N-1} (PSI)	Conv %	% Loss	95% Confidence Level Student T-Test Statistically Significant
<u>Time</u>	<u>Type</u>	<u>Temp</u>					
	CONTROL		237229	5569	2.35%	—	—
4	Ph	RT	233623	6175	2.64%	1.52%	No
4,4	NP	RT	233203	10670	4.58%	1.70%	No
4	NP	RT	231372	7318	3.16%	2.47%	No
8	NP	110°F	230124	4058	1.76%	3.00%	Yes
8	Ph	RT	228408	9267	4.06%	3.72%	No
4,4	Ph	RT	227304	5690	2.50%	4.18%	Yes
8	NP	RT	226987	6746	2.97%	4.32%	Yes
4	NP	110°F	225161	11078	4.92%	5.09%	No
8	Ph	110°F	224460	3056	1.36%	5.38%	Yes
4,4	NP	110°F	224025	1310	0.58%	5.57%	Yes
4,4	Ph	110°F	223598	4220	1.89%	5.75%	Yes
4	Ph	110°F	222890	5582	2.50%	6.04%	Yes

TABLE II(b). 180°F TENSILE TEST RESULTS

Exposure Condition			Average Tensile Strength (PSI)	σ_{N-1} (PSI) Standard Deviation	Coefficient of Variation	% Change from Control	95% Level Statistical Significance
Time	Type	Temp					
4,4	P	110°F	27243	271	0.995%	+6.48%	Yes
4	P	110°F	26854	600	2.23%	+4.96%	Yes
4,4	NP	110°F	26721	707	2.65%	+4.44%	Yes
4	NP	110°F	26700	186	0.70%	+4.36%	Yes
8	P	RT	26552	372	1.40%	+3.78%	Yes
4,4	NP	RT	26311	667	2.53%	+2.90%	No
4,4	NP	RT	26311	624	2.37%	+2.86%	No
8	NP	RT	26179	578	2.21%	+2.32%	No
4	P	RT	26142	695	2.66%	+2.18%	No
4	NP	RT	25853	630	2.44%	+1.05%	No
CONTROL			25585	612	2.39%	—	—
8	P	110°F	25418	375	1.48%	-0.65%	No
8	NP	110°F	24250	350	1.44%	-5.22%	Yes

*Student T-Test @ 95% Confidence Level for Determining if two Populations are Different.

TABLE I. EXPOSURE CONDITIONS

Exposure Time (Hrs.)	Exposure Temperature			
	Room Temperature		110°F	
4	P	NP	P	NP
8	P	NP	P	NP
4,4*	P	NP	P	NP

P = MIL-R-81294 Type I Phenolic

NP = MIL-R-81294 Type II Nonphenolic

Room Temperature = $72 \pm 5^\circ\text{F}$ ($22 \pm 3^\circ\text{C}$)

*4 Hour Exposure, Water Rinse, and an Additional 4 Hour Exposure

TABLE II(a). ROOM TEMPERATURE TENSILE TEST RESULTS

Exposure Condition			Average Tensile Strength (PSI)	σ_{N-1} (PSI) Standard Deviation	Coefficient of Variation	% Change from Control	95% Level Statistical Significance
Time	Type	Temp					
4,4	NP	110°F	23564	165	0.70%	+1.83%	Yes
4,4	Ph	110°F	23431	160	0.68%	+1.25%	Yes
4	Ph	110°F	23312	137	0.59%	+0.74%	Yes
4	NP	110°F	23304	132	0.57%	+0.70%	No
4	Ph	RT	23212	145	0.62%	+0.31%	No
CONTROL			23141	137	0.59%	—	—
8	NP	RT	23114	154	0.67%	-0.12%	No
4	NP	RT	23023	164	0.71%	-0.51%	No
4,4	P	RT	22981	344	1.50%	-0.69%	No
4,4	NP	RT	22881	409	1.79%	-1.12%	No
4	P	RT	22725	133	0.59%	-1.80%	Yes
8	NP	110°F	22722	232	1.02%	-1.81%	Yes
8	P	110°F	22707	270	1.19%	-1.88%	Yes

*Student T-Test at 95% Confidence Level for Determining if Two Populations are Different.

DISTRIBUTION LIST (Continued)

REPORT NO. NADC-85003-60

No. of Copies

NAVAIRDEVCON	8
(3 copies for Code 8131)	
(5 copies for Code 6062)	

DISTRIBUTION LIST

REPORT NO. NADC-85003-60

AIRTASK NO. 4F61-542-000
WORK UNIT ZM540

	<u>No. of Copies</u>
Naval Air Systems Command	8
Washington, DC 20361	
(2 copies for 00D4)	
(1 copy for AIR-31A)	
(2 copies for AIR-5304)	
(1 copy for AIR-5304C)	
(2 copies for AIR-004D4)	
Naval Air Rework Facility (Code 340)	1
Naval Air Station	
Alameda, CA 94501	
Naval Air Rework Facility (Code 340)	1
Naval Air Station	
Jacksonville, FL 32212	
Naval Air Rework Facility (Code 340)	1
Naval Air Station	
Norfolk, VA 23511	
Naval Air Rework Facility (Code 340)	1
Naval Air Station, North Island	
San Diego, CA 92135	
Naval Air Rework Facility (Code 3401)	1
Naval Air Station	
Pensacola, FL 32508	
Naval Air Rework Facility (Code 340)	1
Marine Corps Air Station	
Cherry Point, NC 28533	
Commander Naval Air Force (Code 528)	1
U.S. Atlantic Fleet	
Norfolk, VA 23511	
Commander Naval Air Force (Code 7412)	1
U.S. Pacific Fleet	
Naval Air Station, North Island	
San Diego, CA 92135	
Defense Technical Information Center	12
Cameron Station Bldg #5	
Alexandria, VA 22314	

END

FILMED

10-85

DTIC